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STABILIZATION OF A MAGNETIC FIELD
FOR DETECTION OF NUCLEAR MAGNETIC RESONANCES

A THESIS

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STABILIZATION OF A MAGNETIC FIELD FOR

DETECTION OF NUCLEAR MAGNETIC RESONANCES

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Date Approved by Chairman: April 13, 1961

PREFACE

The magnet power supply described in this thesis was designed and constructed to facilitate the study of nuclear magnetic resonance spectra. The author would like to express his appreciation to Drs. T. L. Weatherly and J. Q. Williams and Mr. E. R. Flynt for the many hours of constructive consultation which they provided.

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SUMMARY

The object of this experimental investigation was to design, construct, and use a magnet power supply for operation of an electromagnet in a nuclear magnetic resonance spectrograph. Also studied was the phenomenon of nuclear magnetic resonance.

Nuclear magnetic resonance is characterized by the emission or absorption of electromagnetic radiation associated with changes in the magnetic quantum number of certain nuclei in the presence of two magnetic fields, one fixed and the other, normal to the first, varying at the resonant frequency. The selection rule governing transitions is the same as for the atomic Zeeman effect. The resonance frequency condition is given by

$$\nu = \frac{g \, \mu_N \, H_o}{h}$$

where h is Planck's constant, g is the spectroscopic splitting factor, $\mu_N = 5.049 \times 10^{-24} \frac{\text{erg}}{\text{gauss}}$ and is the nuclear magneton, and H_o is the fixed magnetic field. The resonance frequency is of the order of 10 megacycles for an applied field of 5,000 gauss.

The sample to be studied is placed in the tank coil of a marginal oscillator. The coil is placed between the pole pieces of a large electromagnet. When the resonance condition is satisfied for the sample nuclei, the nuclei absorb some of the energy from the oscillator coil, thus changing the Q of the circuit and the level of oscillation. The change is

detected by grid rectification. The oscillator frequency is swept at an audio rate by a vibrating capacitor in the tuned circuit, thus the oscillator frequency sweeps across the resonance at an audio rate. The absorption is displayed against the audio sweep on an oscilloscope. For more sensitive detection and a permanent record of the resonance, a phase sensitive detector and chart recorder are used to display the absorption line.

The fixed magnetic field required for observation of nuclear magnetic resonances must be as stable and as homogeneous, over the sample, as possible. The variation of the field cannot be more than a few gauss over the sample volume or the resonance will not be observed. The magnet power supply output is required to have a maximum rate of variation of one part in 100,000 per minute to make the field variation due to magnet current fluctuations much less than the limit placed by the magnet on the field inhomogeneity.

The magnet power supply was designed to make use of a 3.5 kilowatt D.C. generator as the basic power source to provide the 20 amperes at 108 volts needed for maximum magnet power. The power supply has two regulator units. There is a precision regulator of the series type which directly controls the current which is supplied to the magnet. The other regulator unit controls the output of the generator, by varying the field current, in such a manner as to always keep the precision regulator within its operating range.

The stability of the magnetic field was determined by observing the absorption spectra of protons in a 0.5 cubic centimeter sample of water which contained a small amount of ferric nitrate. The fastest rate of change of field was found to be about one part in 100,000 per minute. The total change in field in a particular 10 minute period was about two parts in 100,000. The total variation of field in a five hour period was found to be about one part in 100,000.

CHAPTER I

INTRODUCTION

The magnet power supply described in this study will be used in conjunction with a nuclear magnetic resonance spectrograph for the observation of the radio frequency absorption spectra of certain nuclei. Nuclear magnetic resonance is characterized by the emission or absorption of electromagnetic radiation associated with changes in the orientation of nuclei in a magnetic field.

Suppose an atomic nucleus has an angular momentum, $\vec{M}$, and associated with it is a magnetic moment, $\vec{\mu}$. The angular momentum and magnetic moment are related by

$$\vec{\mu} = g\left(\frac{e}{2m_p c}\right)\vec{M} \tag{1}$$

where e and m_p are the charge and mass respectively of a proton, c is the velocity of light, and g is the spectroscopic splitting factor.

The term "nuclear spin" refers to the largest observable value of the component of M in units of Planck's constant (divided by 2π), $\hbar$, along any specified direction and is represented by I, which is a pure number. The specified direction may be that of a magnetic field $\vec{H}_o$, in which case the observable components of $\vec{M}$ along $\vec{H}_o$ are designated by M_H. The angular momentum and the spin are related by

$$M = \hbar[I(I+1)]^{1/2} \tag{2}$$

The component M_H may take on only the discrete values given by

$$M_H = m_I \hbar \tag{3}$$

where m_I is the magnetic quantum number and may have any of the values I, (I-1), ... , -(I-1), -I. The relationships between some of these quantities are shown in Figure 1.

Corresponding to this quantization of the angular momentum component, the nuclear magnetic moment also has (2I+1) components in proportion. The components of $\vec{\mu}$ along the applied field $\vec{H}_o$ are

$$\mu_H = g\left(\frac{e}{2m_p c}\right)\hbar\, m_I = g\, \mu_N\, m_I \tag{4}$$

where $\mu_N = 5.049 \times 10^{-24}$ erg/gauss is called the nuclear magneton.

An applied magnetic field produces a splitting of the energy levels. Since the energy of a dipole $\vec{\mu}$ in a magnetic field is equal to $- \mu_H H_o$, there will be as many energy levels as there are possible components of $\vec{\mu}$ in the direction of the field. These levels are illustrated in Figure 1 for the case of I = 3/2, and in the general case show a set of equally spaced levels with separation $g\, \mu_N\, H_o$ between successive levels.

The selection rule governing transitions between energy levels is the same as for the closely related atomic Zeeman effect; transitions are allowed which cause m to change by $\pm$ 1. This means that transitions are possible only between adjacent levels. A quantum of energy of angular frequency ω can therefore excite transitions between the energy levels if it has the same magnitude as the level spacing, that is

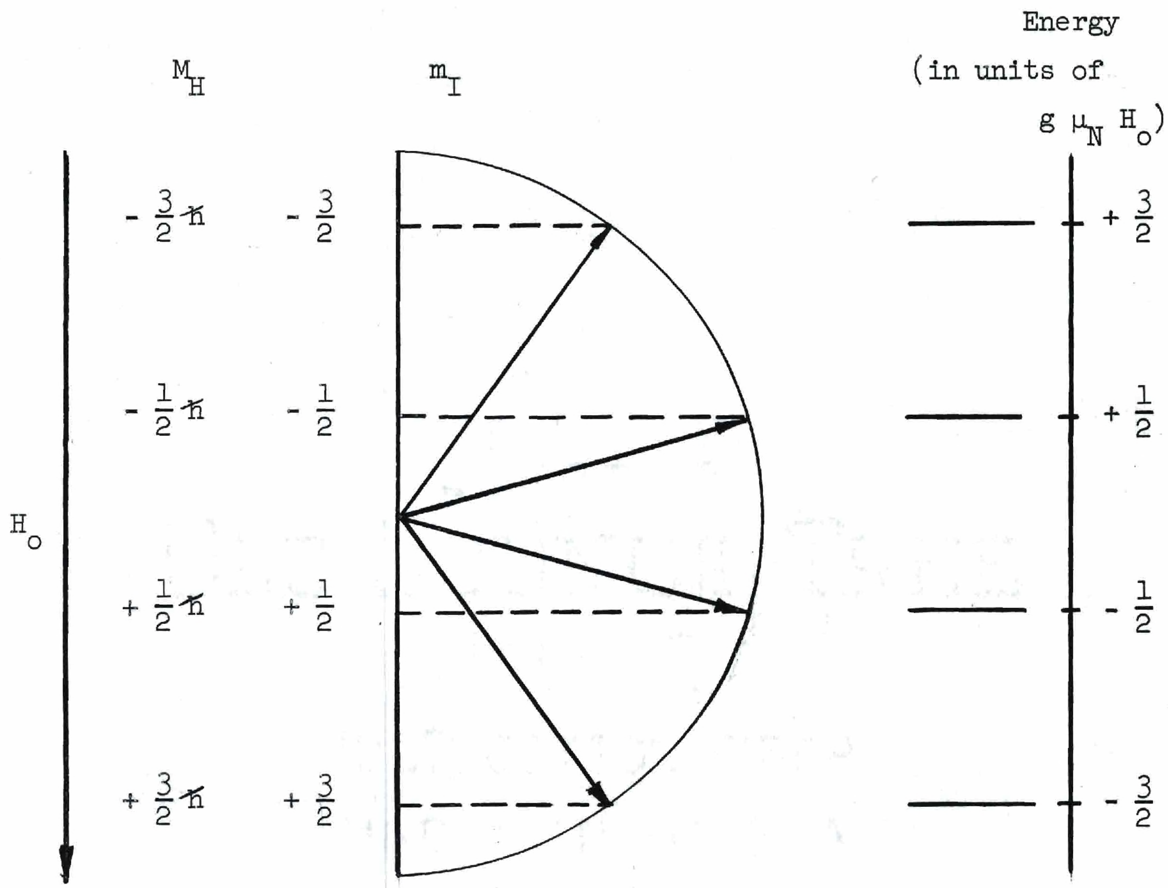


Figure 1. Observable Values of Energy and Component of Angular Momentum Along Magnetic Field Direction for a Nucleus with I = 3/2.

$$h\omega = g\ \mu_N\ H_o\ . \tag{5}$$

One can determine the spectroscopic splitting factor, g, by measuring the frequency of the exciting radiation and the field intensity, H_o. Equation (4) then gives $g\ \mu_N\ I$ for the maximum value of μ_H. This maximum value of μ_H is called the nuclear magnetic moment, and tables of nuclear magnetic moments list values of this quantity rather than that given by equation (1).

In conventional optical spectroscopy emitted radiation is analyzed, whereas in the nuclear magnetic resonance absorption experiment radiation is generated externally and its effect on the atomic system is investigated. However, the necessary condition for observation of the magnetic resonance transitions may be found from the properties of the emission spectrum of the atomic Zeeman effect. Zeeman effect transitions which involve a change in M of $\pm$ 1 produce radiation which is circularly polarized in the plane perpendicular to the steady magnetic field $\vec{H}_o$. In order to excite such transitions in the magnetic resonance experiment, it is therefore necessary to supply radiation with a circularly polarized component of the magnetic vector in a plane perpendicular to the steady magnetic field.

This requirement of circular polarization is just what one would expect by classical arguments. If a magnetic dipole $\vec{\mu}$ is placed in a magnetic field $\vec{H}_o$ as shown in Figure 2, the dipole precesses about the direction of the magnetic field. The rate of precession is given by the Larmor angular frequency

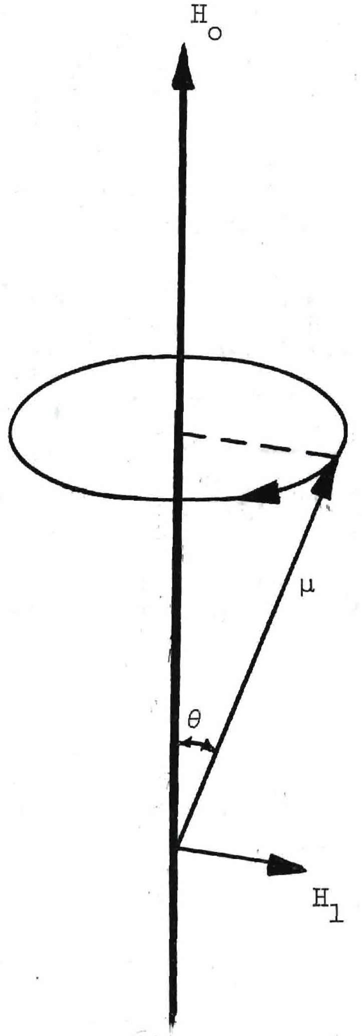


Figure 2. Classical Larmor Precession.

$$\vec{\omega}_{\ell} = \gamma \, \vec{H}_{o} \, , \tag{6}$$

where $\gamma = \frac{g \, \mu_N}{h}$ is the magnetogyric ratio of the dipole. The Larmor frequency is derived from purely classical considerations in the Appendix. For a field of 10,000 gauss, the Larmor frequency of a nucleus is on the order of ten megacycles.

It is possible to generate a circularly polarized electromagnetic field but usually it is much simpler to produce a linearly oscillating field. A linearly oscillating field will do the job here as it can be considered to be the superposition of two rotating fields. Thus if the linearly oscillating field has amplitude $2H_1$, it may be decomposed into two circularly polarized fields, each of amplitude H_1 and rotating with frequency ω_{ℓ}, but rotating in opposite senses in a plane perpendicular to $\vec{H}_o$. Resonance will be obtained with the component which has the correct sense. It is not generally possible to tell which component is utilized, and it is not usually necessary to know this. A complete discussion of nuclear magnetic resonance can be found in the book by E. R. Andrew (1).

Now consider an assembly of identical atomic nuclei in the presence of a steady magnetic field $\vec{H}_o$. This is purely a logical extension of the problem of one nucleus as already discussed. For simplicity, at first suppose that the nuclear spin number is 1/2. If it is assumed that there is only a weak coupling between the nuclear magnets, then, to a first approximation, the magnetic interaction between the nuclei may be neglected. This means that the energy levels discussed earlier for the single nucleus will be the same as for each nucleus of the assembly.

There must, however, be some coupling between the nuclei so that the assembly may be considered to be in thermal equilibrium at a temperature T_s.

If it is supposed here that the interaction of the nuclear magnets with the remainder of the system is even smaller than the coupling between nuclei, it will be possible to calculate the relaxation times of the nuclear system by more or less elementary considerations. In other words, for these assumptions to hold, the material must be diamagnetic except for a feeble paramagnetism produced by the nuclei. A detailed development of the concept of relaxation time may be found in the paper by Bloembergen, Purcell, and Pound (2).

Since $I = 1/2$, each nucleus has two possible energy levels separated by a gap of $2\mu_H H_o$. If now radiation of the resonance frequency is applied, polarized in a direction perpendicular to H_o, transitions between the two levels take place. From statistical theory it is known that the probability of transition upward by absorption is equal to the probability of transition downward by stimulated emission. If the numbers of nuclei in each energy level were equal, the average rate of transitions up and down would therefore be equal, and there would be no net effect on the system. Actually, however, since the nuclear spins are in equilibrium at temperature T_s, the ratio of the population of the lower level to that of the upper level is given by the Boltzmann factor

$$\frac{N_{lower}}{N_{upper}} = e^{\frac{2\,\mu_H\,H_o}{k\,T_s}} \qquad (7)$$

where k is Boltzmann's constant. Because of the small excess of population in the lower energy state, there is a net absorption of energy from the radio frequency field.

The absorption of energy corresponds to the transfer of some of the excess population in the lower level to the upper level. If there is no interaction between the system of nuclear spins and the lattice, the fractional excess of population,

$$\frac{N_{lower} - N_{upper}}{N_{lower}} = \frac{2\,\mu_H\,H_o}{k\,T_s} \tag{8}$$

steadily decreases. The ratio of the populations may still formally be described by a Boltzmann factor, with the temperature T_s of the spin system steadily rising. The spin system is in fact being subjected to a process of radio frequency heating. The temperature of the lattice is not affected, however, since there was assumed negligible interaction between the spin system and the lattice.

In any actual physical system the nuclear spins do interact with the lattice but this interaction is often very small. It is only because of the assumption that spin-lattice interaction is small that it is possible to separately consider a spin temperature and a lattice temperature. Such interaction as there is between the two systems tend to bring both into thermal equilibrium at the same temperature. This common temperature is almost identical with the lattice temperature, since except at extremely low temperatures the heat capacity of the spin system is very small compared with that of the lattice. Thus, while the radio frequency radiation is reducing the excess of population in the lower energy state, the interactions with the lattice are tending to restore the excess to its original value.

Suppose the spin system and lattice system which are in thermal equilibrium are disturbed by the brief application of a radio frequency field or by a change in the steady magnetic field. The spin populations adjust themselves, by interaction with the lattice, to the values required by thermal equilibrium with some spin-lattice relaxation time, T_1. This can be shown by a fairly simple argument.

In order that the spin system may cool down to the temperature of the lattice after removal of the perturbation there must be a net downward transfer of nuclei to the lower energy level. Since the population of the upper level does not exceed that of the lower level, this can only be possible if the probability for downward transitions W_- exceeds that for upward transitions W_+. This does not contradict the statement of equality that was made earlier as it is the result of a different interaction.

If the whole system is in thermal equilibrium at a temperature T, then the number of transitions upward and downward must be equal:

$$W_+N_+ = W_-N_- \tag{9}$$

where N_- is the number of nuclei in the upper energy state ($m = -1/2$) and N_+ is the number of nuclei in the lower state ($m = +1/2$). N_+ and N_- are related by the Boltzmann factor, so that

$$\frac{W_-}{W_+} = \frac{N_+}{N_-} = e^{\frac{2\,\mu_H H_o}{kT}} \simeq 1 + \frac{2\,\mu_H H_o}{kT} \tag{10}$$

Thus

$$W_- = W(1 + \frac{\mu_H H_o}{kT}) \tag{11}$$

and

$$W_+ = W(1 - \frac{\mu_H H_o}{kT}) \tag{12}$$

where W is the mean of the two transition probabilities W_+ and W_-.

Now consider the spin system at a temperature T_s different from the lattice temperature T, and remembering that the excess number of nuclei, n, in the lower state, where

$$n = N_+ - N_- \tag{13}$$

changes by 2 with each transition, then the rate of change of n is

$$\frac{dn}{dt} = 2N_-W_- - 2N_+W_+ \tag{14}$$

By using the values found earlier for W_+ and W_-, the rate of change of n becomes

$$\frac{dn}{dt} = 2W(n_o - n) \tag{15}$$

where

$$n_o = \frac{N\,\mu_H H_o}{kT} \tag{16}$$

and

$$N = N_+ + N_- \tag{17}$$

is the total number of nuclei. Thus n_o is the value of n when the spin system is in thermal equilibrium with the lattice. By integration

$$n_o - n = (n_o - n_a)e^{-2Wt} \tag{18}$$

where n_a is the initial value of n. Thus equilibrium is approached exponentially with a characteristic time given by

$$T_1 = \frac{1}{2W} \tag{19}$$

which may be called the spin-lattice relaxation time or the thermal relaxation time. The values of T_1 found experimentally usually lie in the range 10^{-4} to 10^{4} seconds.

Now consider the effect of the interaction between the spins themselves. Since each nucleus possesses a small magnetic dipole moment there will be a magnetic dipole-dipole interaction between each pair of nuclei. From a classical point of view this may be regarded in the following way. Each nuclear magnet finds itself not only in the applied steady magnetic field $\vec{H}_o$, but also in the small local magnetic field $\vec{H}_{local}$ produced by the neighboring nuclei. The direction of the local field differs from nucleus to nucleus, depending on the relative disposition of the neighboring nuclei and on their magnetic quantum number m.

The magnetic field of a magnetic dipole of moment μ at a distance r is of the order

$$H_{local} \sim \frac{\mu}{r^3} \tag{20}$$

or about 5 gauss for typical values of μ and r. The resultant magnetic field will not then be the same for each nucleus, but will vary from nucleus to nucleus. It follows that the resonance condition will not be as sharp as predicted by the simple case earlier but will be broadened by an amount of approximately $g\,\mu_H\,H_{local}$. Since the resultant field differs from nucleus to nucleus, there will be a distribution of the frequencies of their Larmor precession, covering a range of $\delta\omega_o$, given by

$$|\delta\omega_o| = |\omega_o' - \omega_o| \doteq |\gamma(\vec{H}_o + \vec{H}_{local})| - |\gamma\vec{H}_o| \tag{21}$$

which gives

$$\delta\omega_o \sim \gamma H_{local} \sim \frac{\mu_N^2}{\hbar\, r^3} \tag{22}$$

since

$$\gamma \sim \frac{\mu_N}{\hbar}\,. \tag{23}$$

Thus if two spins have precession frequencies differing by $\delta\omega_o$ and are initially in phase, then they will be 180° out of phase in a time

$$T_2 \sim \frac{1}{\delta\omega_o} \tag{24}$$

where T_2 is called the spin-spin interaction time.

This is not the only way in which the relative phases of two precessing spins j and k may be disturbed. Nucleus j produces at nucleus k a magnetic field oscillating at its Larmor frequency, and as a result may induce a transition in k. The energy for the transition of nucleus k comes from nucleus j. Since the relative phases of the nuclei change 180° in a time of the order of $\frac{1}{\delta\omega_o}$, the correct phasing for this spin-exchange process should occur after a time interval of this order, and this in turn should determine the lifetime of a spin state. A lifetime of $\frac{1}{\delta\omega_o}$ produces a broadening of the energy levels of the order of $\hbar\delta\omega$, thus

$$\hbar\delta\omega_o \sim \hbar\gamma\, H_{local} = g\, \mu_N\, H_{local} \tag{25}$$

gives the value of the energy level broadening.

These two phase-disturbing and line-broadening processes are both present only where identical nuclei are concerned. For a system of non-identical nuclei, the local field effect is still present, but the spin-exchange process is absent, since the Larmor precession frequencies of the nuclei are very different.

Consider now the possibility of saturation of the spin system by the applied fields. In absence of radiation, the differential equation governing the time variation of the excess number n of nuclei in the lower state is found to be

$$\frac{dn}{dt} = 2W(n_o - n) = \frac{n_o - n}{T_1} \tag{26}$$

from equations (15) and (19). When radiation is present another term must be added to account for the upward transitions which correspond to the net absorption of energy. Thus

$$\frac{dn}{dt} = \frac{n_o - n}{T_1} - 2nP \tag{27}$$

where P is the probability per unit time of a transition by a nucleus between the two levels under the influence of the radiation. Again it must be noted that each upward transition reduces n by 2. A steady state is reached when $\frac{dn}{dt} = 0$, and in this condition the steady state value, n_s, of the excess number is given by

$$\frac{n_s}{n_o} = \frac{1}{1 + 2PT_1} . \tag{28}$$

It is now necessary to evaluate the transition probability P. If a nucleus is subjected to a radio frequency magnetic field of amplitude H_1 rotating in the correct sense in a plane perpendicular to $\vec{H}_o$, the probability of a transition in unit time between states designated by magnetic quantum numbers m and m' is found by radiation theory to be (2)

$$P_{m \to m'} = \frac{1}{2} \gamma^2 H_1{}^2 |\langle m|I|m'\rangle|^2 g(\nu) \tag{29}$$

where $\langle m|I|m'\rangle$ is the appropriate matrix element of the nuclear spin operator and $g(\nu)$ is the frequency shape function of the absorption line. The spin-spin relaxation time and the frequency shape function are related by (2)

$$T_2 = \frac{1}{2} g(\nu)_{max} , \tag{30}$$

because $g(\nu)$ and T_2 both are measures of the frequency spread associated with the absorption line width.

The non-diagonal matrix elements $\langle m|I|m'\rangle$ are zero except when $|m-m'|=1$, corresponding to the selection rule mentioned earlier. For $m'=m-1$,

$$|\langle m|I|m'\rangle|^2 = \frac{1}{2}(I+m)(I-m+1) \tag{31}$$

and thus

$$P_{m\to m-1} = \frac{1}{4}\gamma^2 H_1{}^2 g(\nu)(I+m)(I-m+1) \ . \tag{32}$$

For the case $I = \frac{1}{2}$, this reduces to

$$P = \frac{1}{2}\gamma^2 H_1{}^2 g(\nu) \tag{33}$$

and thus

$$\frac{n_s}{n_o} = \frac{1}{1 + \frac{1}{2}\gamma^2 H_1{}^2 T_1 g(\nu)} \equiv Z \ . \tag{34}$$

If a radio frequency field is applied, whose amplitude H_1 is large, $\frac{n_s}{n_o}$ becomes quite small; the spin temperature T_s becomes very high, and the spin system is said to be saturated. For this reason the expression Z is called the saturation factor. Saturation is greatest at the frequency which gives $g(\nu)$ its maximum value. Thus the saturation factor has the value

$$Z_o = \frac{1}{1+\gamma^2 H_1^{\,2} T_1 T_2} \tag{35}$$

Since

$$n_o = \frac{N\mu_H H_o}{kT} , \tag{16}$$

then characterizing n_s with a spin temperature T_s,

$$n_s = \frac{N\mu_H H_o}{kT_s} , \tag{36}$$

and by combining these two expressions to find the spin temperature:

$$T_s = T \frac{n_o}{n_s} = \frac{T}{Z} . \tag{37}$$

The spins can readily be heated up to extremely high temperatures, and it does not require very much energy from the radio frequency field to accomplish this heating. In fact, to raise the spin temperature from that of the lattice, where the excess population of the lower state is n_o, to an infinite temperature, where the excess is zero, energy must be supplied equal to $\frac{n_o \mu_H H_o}{2}$. Substituting for n_o, the energy becomes

$$E_{sat} = \frac{N(\mu_H H_o)^2}{2kT} \tag{38}$$

For example, T_1 for protons in ice in a field of 7000 gauss at 88°K is about 10^4 seconds, while T_2 is about 10^{-5} seconds. If the system is subjected to a radio frequency field $H_1 = 0.1$ gauss, a spin temperature of about $10^8\,°K$ may be obtained. The increase in energy is only about 1 erg per mole, however.

A detailed discussion of the applications of nuclear magnetic resonance may be found in Andrew (1). The most obvious application is to the determination of nuclear properties. From the resonance condition one may obtain with considerable accuracy values of the nuclear magnetogyric ratio γ for any stable nucleus of reasonable abundance. The nuclear spin number can often be found if it is not already known, and hence the nuclear magnetic moment is obtained. The sign of the nuclear moment is also obtainable. Nuclei with spin number $I > \frac{1}{2}$ have an electric quadrupole moment, relative values of which may be determined from the fine structure of the nuclear magnetic resonance spectrum. Once a nuclear magnetogyric ratio is known, it may be used for the calibration of magnetic fields, and for related problems with magnets. The great precision of measurement of the nuclear magnetic resonance frequency in a given magnetic field has led to the accurate determination of certain important physical quantities.

The nuclear magnetic resonance has been observed in matter in all its forms: liquids and gases, ionic and molecular solids, and metals. In each case, and particularly for the solid state, valuable information has been obtained concerning the structure and other non-nuclear properties of the material, through the effects of environment on the resonance spectra.

The resonance spectrum for liquids provides a tool for chemical analysis and identification. Changes in this spectrum can be used to follow the progress of certain chemical reactions. Information can also be obtained concerning self-diffusion coefficients in liquids and the formation of complexes. In some cases the water content of biological materials may be measured.

The resonance spectrum of non-metallic solids frequently provides structural information, and can lead to knowledge of hindered rotation of molecules or groups in the crystal. Self-diffusion may be studied both in non-metals and in metals. The protons in hydrated paramagnetic and anti-ferromagnetic crystals provide magnetic indicators for study of the magnetic properties of these crystals. In the case of metals an interaction between the nuclei and the conduction electrons leads to information concerning the electronic states at the top of the Fermi distribution. The resonance spectrum sometimes provides information concerning short-range order in alloys, and concerning defects in both metals and non-metallic solids.

CHAPTER II

THE NUCLEAR MAGNETIC RESONANCE SPECTROGRAPH

The first successful nuclear magnetic resonance experiments using bulk material were carried out independently at the end of 1945 by Purcell, Torrey and Pound (3) and by Bloch, Hansen and Packard (4). Purcell and his colleagues found first the resonance absorption for the protons in solid paraffin, and detected it by measuring the additional loss which it caused in the tuned circuit supplying the radio frequency power. For this purpose the tuned circuit was placed in one arm of a balanced radio frequency bridge. Bloch and his colleagues found their first resonance for the protons in water. Their method of detection was a novel one. The reorientation of the nuclear moments by the resonant electromagnetic field induces an electromotive force at the resonant frequency in a coil whose axis is perpendicular to the steady magnetic field. Bloch called this method of detection nuclear induction.

Since the original experiments in nuclear magnetic resonance many other techniques have been developed (1). For the spectrometer described here the marginal oscillator method is used because of its simplicity and its ability to rapidly search a wide frequency range. The circuit used is patterned after the autodyne of N. J. Hopkins (5) with a modification which was due to R. Livingston (6). A block diagram of the spectrometer is shown in Figure 3 along with a schematic diagram of the oscillator in Figure 4. The phase-sensitive detector indicated in Figure 3 was designed

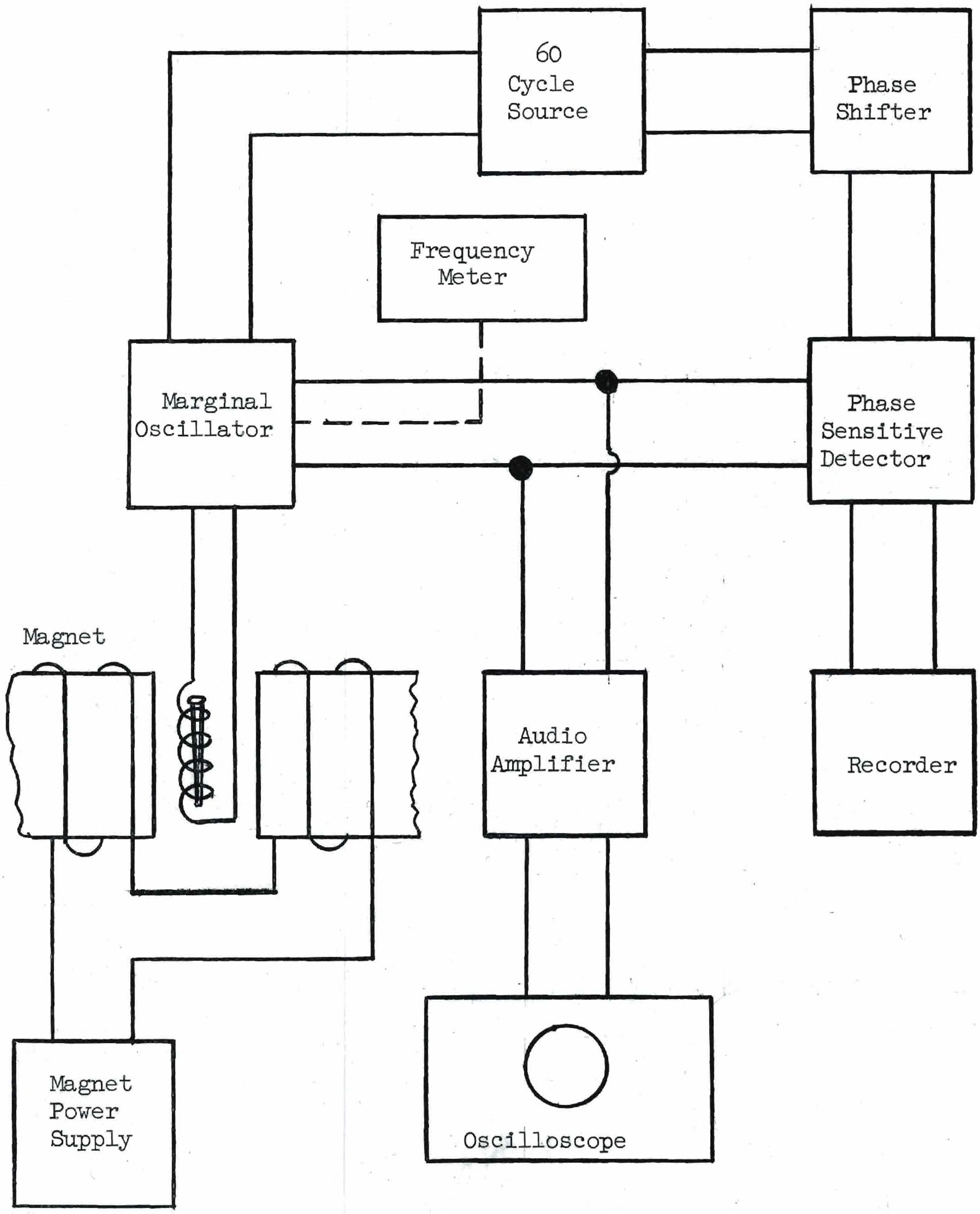


Figure 3. Block Diagram of Spectograph.

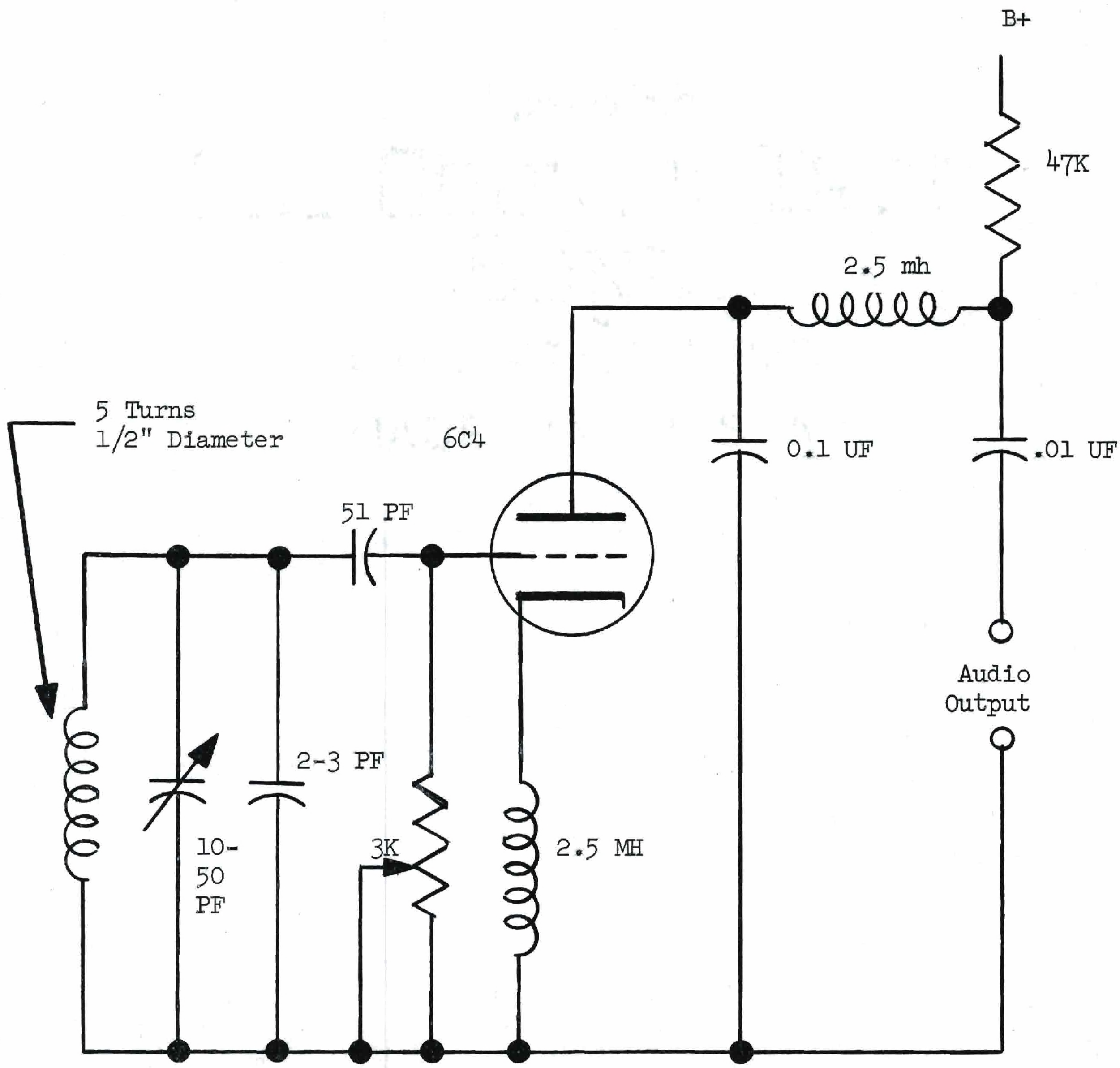


Figure 4. Schematic Diagram of the Marginal Oscillator.

and constructed by E. H. Davidson (7). The magnet is a Diecraft Model One electromagnet with seven inch diameter pole pieces. The marginal oscillator uses grid rectification and is swept in frequency by a vibrating reed capacitor which is driven from the 60 cycle A.C. line. The frequency of the oscillator may be varied between 20 megacycles and 40 megacycles by a worm-gear driven capacitor. The frequency is monitored by means of a L M 7 frequency meter which is similar to a B.C. 221.

Changes in the plate current of the oscillator are amplified and viewed on an oscilloscope when one is making a rapid search for resonances. The audio amplifier contains two twin-tee filters to remove the incidental 60 and 120 cycle amplitude modulation due to the sweep arrangements. Weak signals require the use of the phase-sensitive detector and paper tape recorder. The magnet power supply provides the necessary magnet current with sufficient stability for observable resonant absorption. The power supply also provides a slow current sweep for use with the phase-sensitive detector and recorder. The magnet power supply was designed and built for use with this spectrograph. The other units were already available.

The sample is contained in a cylindrical coil placed with its axis perpendicular to the direction of a steady magnetic field. This coil and a condenser form a parallel tuned combination in the grid circuit of a marginal oscillator. An oscillator is an amplifier with sufficient positive feedback to supply its own input, and thus maintain continuous oscillation. Formally, a regenerative amplifier may in fact be regarded as the placing of a negative resistance in parallel with the tuned circuit, so that if this negative shunt resistance more than compensates the positive shunt resistance of the tuned circuit, oscillations are sustained. The amplitude of the oscillations builds up until the average value of the negative resistance over a cycle, as determined by the curvature of the tube characteristic, just equals the positive shunt resistance. Radiofrequency current thus flows in the sample coil, and when the nuclear magnetic resonance condition is reached an absorption of radiofrequency energy occurs, thus causing a decrease in the positive shunt resistance. The oscillation amplitude therefore falls every time the modulated 'steady' magnetic field passes through the resonance line. In operation the magnitude of the negative shunt resistance is adjusted to be just small enough to allow oscillations to be sustained. Since the curvature of the tube characteristic is small for such a small voltage swing, the level of oscillation is very sensitive to the small changes in shunt resistance which result from passage through the nuclear resonance condition.

One reason for restricting the oscillation amplitude to a small level is that the sensitivity is then greatest. A second reason is that at higher levels the tube characteristics are more non-linear, so that noise components originating in a wider band of frequencies are mixed with the nuclear absorption signal, thus increasing the noise factor. A more important reason, however, is to prevent saturation of the specimen. Using a marginal oscillator it is not possible, consistent with stable operation, to reduce the radiofrequency amplitude across the specimen coil much below 0.1 volt. While such a level does not generally produce saturation when the specimen is in liquid form, many solids would be saturated. In fact, it is often necessary with solids having a long spin-lattice relaxation time, to work with a radiofrequency level of a few millivolts if saturation is to be avoided; the marginal oscillator method is therefore not suited to the study of such materials.

The audio-modulated radiofrequency voltage appearing across the sample coil is rectified within the oscillator itself by grid rectification. This technique makes the circuit an oscillating detector. The grid rectification assists, moreover, in keeping the amplitude of oscillation at a low level. As the amplitude builds up, grid rectification pushes the operating point back to more negative values of grid voltage where the mutual conductance of the tube, and therefore its gain, is lower.

A stable, homogeneous magnetic field is required for observation of nuclear magnetic resonance. The variation of the "constant" magnetic field cannot be more than a few gauss over the sample volume or the resonance will not be observed. The effects of field fluctuations, due to power supply variations and temperature changes, and the effects of field gradients, due to the finite size of the pole pieces and inhomogeneous magnet materials, are similar in that they increase the resonance line width and reduce the amount of information that may be obtained from observation of the resonance. The field gradients are more or less fixed by the magnet. One can reduce the sample size and use magnet pole pieces with special shapes so as to reduce the inhomogeneity of the field over the sample volume; however, the practical lower limit on inhomogeneity was found to about 0.1 gauss with a spectrometer consisting of the Diecraft electromagnet and marginal oscillator.

The magnet power supply is required to have a stability over the maximum observation period of one minute such that field fluctuations, due to magnet current variations, will be considerably less than 0.1 gauss. This requirement insures that the field homogeneity will be limited only by the magnet. The magnet power supply was designed to make use of a 3.5 kilowatt D.C. generator as the basic power source to provide the 20 amperes at 108 volts needed for maximum magnet power. The requirement of 0.1 gauss or less field variation out of 5000 to 10,000 gauss total field means a current variation of one part in 100,000 or less is required. A current regulator was necessary since the generator does not have the required output stability.

CHAPTER III

DESCRIPTION OF THE MAGNET POWER SUPPLY

The instrument to be described was developed for the purpose of supplying a 5.4 ohm, 0.45 henry electromagnet with a regulated current which is continuously variable between 3 and 25 amperes. The current regulation is 0.001 per cent or better. There is a one cycle/second linear current sweep, with sweep widths variable from 2 milliamperes to 5 amperes, included in the fine regulator unit. Magnet power supplies with characteristics similar to the one described here have been previously described by Johnson and Singer (8) and Garwin, et al., (9).

A block diagram of the magnet power supply is shown in Figure 5. The power supply consists of a 3.5 kilowatt motor generator set and two current regulators. The fine regulator unit consists of the series regulator, reference resistor, reference battery, chopper amplifier, and D.C. amplifier. The voltage drop across a selected portion of the reference resistor is compared with the reference voltage and the error voltage is amplified by the chopper amplifier and D.C. amplifier. The output of the D.C. amplifier drives the series regulator in such a manner as to reduce the error voltage. Since the voltage drop across the reference resistor is directly proportional to the current flowing through the magnet, the magnet current is held constant by the series regulator. A variable portion of the voltage drop across the reference resistor can be compared with the reference voltage, permitting the magnet current to be varied

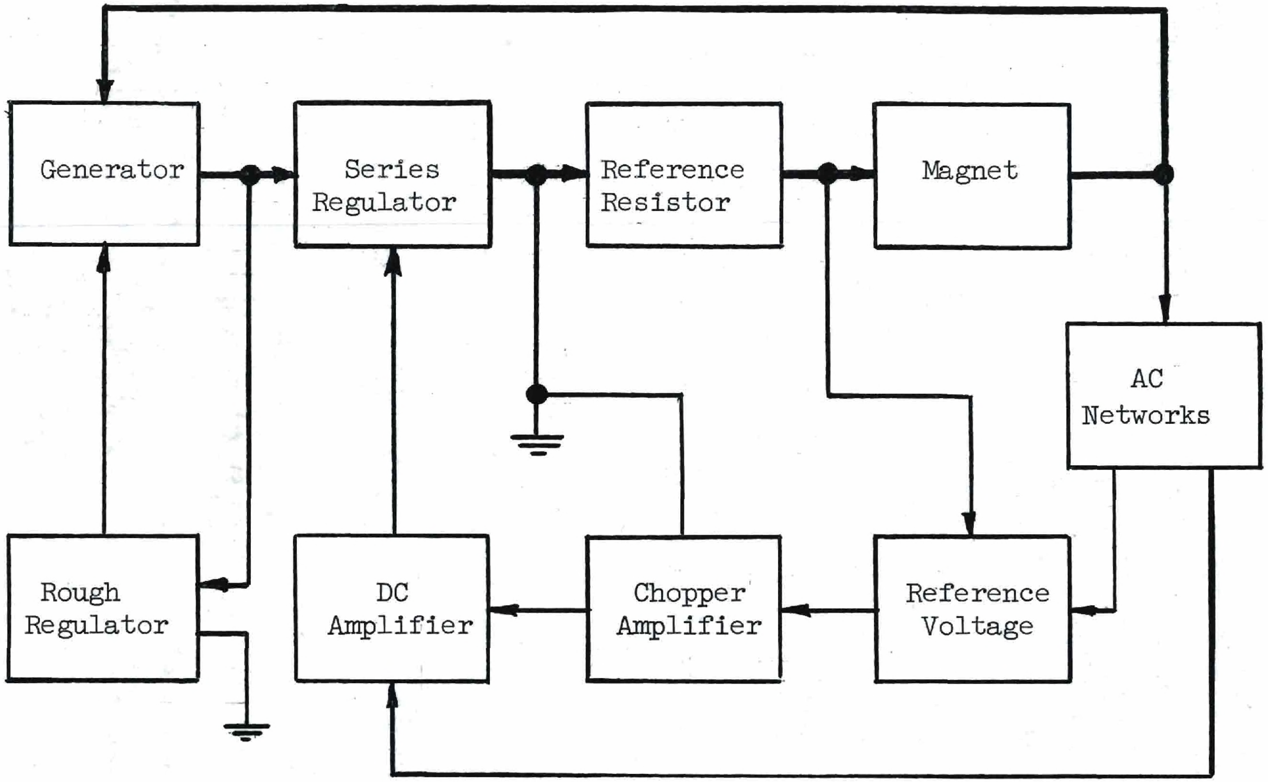


Figure 5. Block Diagram of Magnet Power Supply.

over a wide range. High frequency current fluctuations are sensed as voltage changes across the magnet and are fed back through A.C. networks to the inputs of the chopper amplifier and D.C. amplifier. These A.C. coupled feedback paths insure that the regulator has adequate frequency response. The rough regulator controls the generator field current in such a manner as to keep the voltage drop across the series regulator between four and five volts. This feature is necessary so that the series regulator will be within its rating throughout the whole range of operating currents.

Since this power supply contains negative feedback loops, it was necessary to meet certain stability requirements on gain and phase shift, the primary requirement being that loop gain reach unity before the phase shift reaches 180°. The theory of negative feedback in regulating systems is well established (10) (11). The characteristics of the chopper amplifier and D.C. amplifier were investigated by means of gain _vs._ frequency and phase shift _vs._ frequency plots. The characteristics of the overall system were determined in a general manner from asymptotic gain _vs._ frequency plots of the major time constants of the system. The rough regulator loop stability was investigated in a similar manner. However, the addition of the rough regulator to the power supply increased the complexity considerably, as it increased the number of loops present in the system. Both gain and phase shift of the rough regulator were chosen primarily to satisfy stability requirements as the tightness of the control of the voltage drop across the series regulator is not important. An added complexity arises from the fact that the magnet inductance varies with the current, frequency, and air gap. The complexity and non-linearity made it necessary to make final adjustment of the values of the various corrective networks experimentally.

A schematic diagram of the magnet power supply is shown in Figure 6. The fine regulator is placed in series with the output of a motor generator. Five 2N277 power transistors are paralleled to form the series regulator and can safely dissipate up to 200 watts. The transistors are mounted on a water-cooled copper panel. This copper plate is used as the common ground point of the system. The 0.5 ohm resistors in the emitter leads of the series regulators are used to insure the proper current division between transistors. Additional precautions against unequal loading are the 12 gauge base and emitter buses. The current per transistor is held below 5 amperes so as to maintain high gain because the 2N277 beta begins to fall off above this value.

Current is sensed as the voltage drop across a 1.185 ohm resistor which was fabricated of number 18 constantan wire and immersed in transformer oil. The resistor assembly is cooled by a water jacket. The choice of the reference resistor is a compromise between amplifier gain and power dissipation. The larger the reference resistor's value, the lower the amplifier gain can be for a given degree of regulation. The upper limit is fixed by dissipation in two ways. First, there is the limit set by the generator output and second there is the difficulty of resistance changes due to heating of the reference resistor.

To establish the desired current a reference voltage is compared with a fraction of the potential developed across the reference resistor.

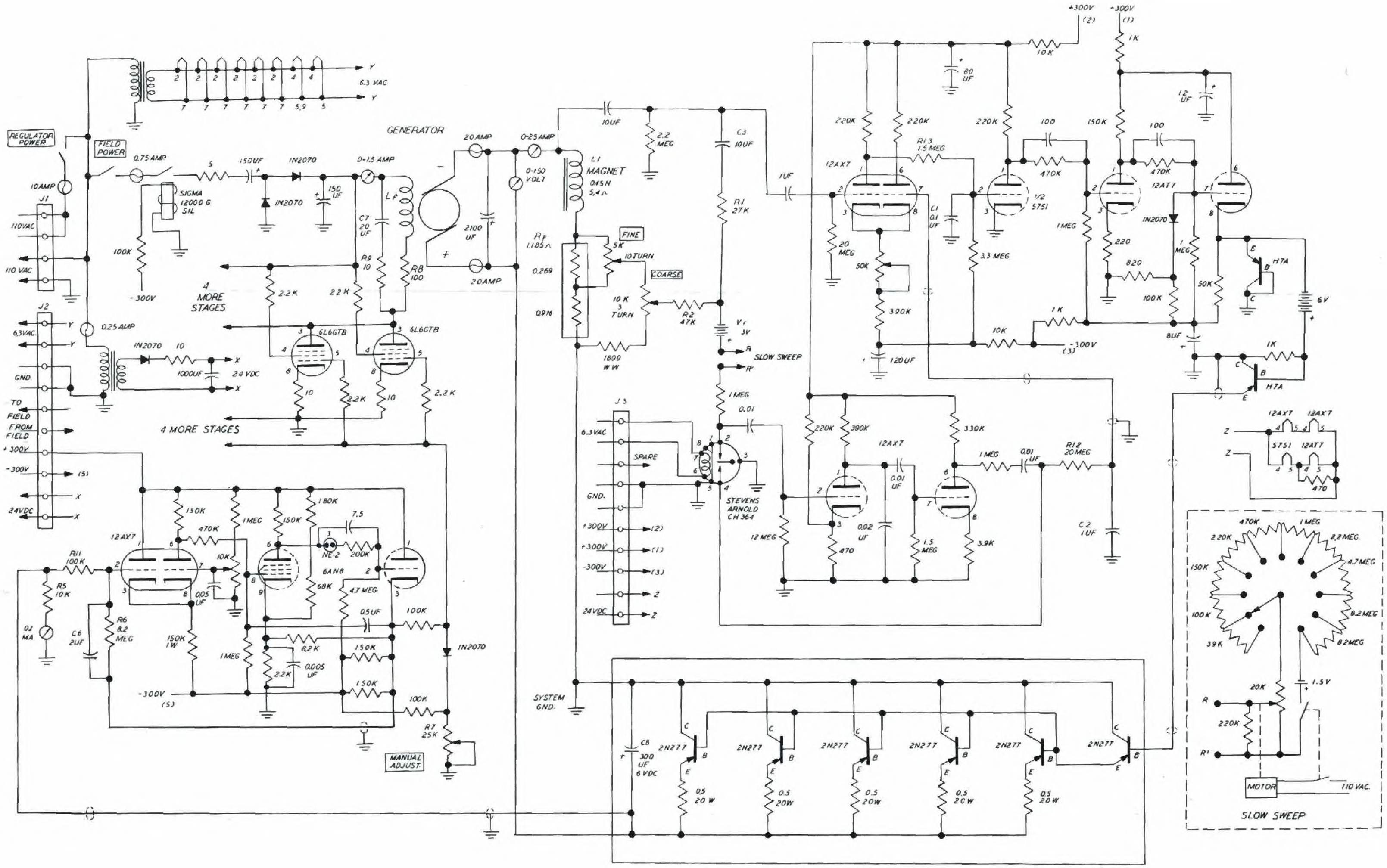


Figure 6. Schematic Diagram of Magnet Power Supply.

The fraction of the potential drop is determined by the settings of two Helipots across the reference resistor. One Helipot is a 5 kilohm, 10 turn, vernier current adjust and the other is a 10 kilohm, 3 turn, coarse current adjust. The difference between the reference voltage and the fraction of the potential that is set by the current controls is amplified by a chopper amplifier, filtered, amplified by a three-stage D.C. amplifier, and fed to the series regulator. The gain of the chopper amplifier and D.C. amplifier is such that a ten microvolt signal at the input of the chopper amplifier is sufficient to give a 20 ampere change in current. However, long term amplifier drift is about 30 microvolts referred to the input of the chopper amplifier. This means that the long term regulation is limited to one part in 10^5 by amplifier drift since the reference voltage is three volts. The regulation is limited to about one part in 10^4 over periods of more than a few hours by the stability of the reference battery.

Although the current control loop in the fine regulator has very high D.C. gain, it has poor response to any A.C. signal whose period is less than a few seconds. To overcome this difficulty and to enable the regulator to remove power supply ripple, two parallel A.C. coupled feedback paths are used to bypass the current control loop and provide high A.C. gain. One A.C. path is the resistor and capacitor network, C_3, R_1, which develops feedback voltage across the resistor, R_2, in series with the current input to the chopper amplifier. The other A.C. feedback path is to one of the grids of the differential stage of the D.C. amplifier. The resistor and capacitor networks, R_3, C_4 and R_4, C_5, have no effect on the phase shift, but serve the function of removing the magnet D.C. voltage from the A.C. feedback paths.

The D.C. amplifier is a three stage amplifier with a cathode follower output. The first stage is a differential amplifier with A.C. input to one grid and the chopper amplifier output to the other grid. The other two stages are conventional triode amplifier stages. The D.C. amplifier contains a diode clamp as protection for the power transistors. A 1N2070 diode is placed in the grid circuit of the cathode follower in such a manner as to prevent the output of the cathode follower from going positive.

Another protective clamp is provided by a H7-A transistor which is connected as a diode and back-biased at 6 volts. If for any reason the voltage across the series regulator transistors should rise, this clamp will act to prevent overvoltage. The transistor current will still rise, but the generator output is fused at 20 amperes to limit the current.

The coarse regulator controls the generator output to maintain the voltage drop across the series regulator transistors within a suitable operating range. The minimum satisfactory drop is about one volt; below this value the gain falls off very sharply. The maximum voltage drop is fixed by dissipation considerations. Since the regulator holds the voltage across the load constant in the face of generator ripple, the operating point must be chosen so as to keep the transistors from being driven below one volt. As the operating voltage is raised, however, dissipation is increased. Therefore, it is desirable to lower the ripple so that less power will have to be dissipated. To smooth the input, a 2100 microfarad filter is connected, with heavy wire, across the generator output. The filter is located in the regulator rack so as to make maximum use of the impedance of the generator leads. The generator output ripple is still two to three volts peak to peak and thus the operating point is chosen to be between 4 and 5 volts. The operating point varies from 5 volts at low power levels to about 4 volts at maximum power.

The voltage drop across the transistors is compared with a reference voltage in the differential first stage of a Philbrick K2-X operational amplifier. The difference voltage is amplified and used to drive the series regulator which controls the generator field current.

The reference voltage is set by means of a screwdriver adjustment, R_{10}, on the rough regulator chassis. The voltage across the transistors is determined with the aid of a 0-10 volt voltmeter which is mounted on the rough regulator chassis.

The operational amplifier is a high gain D.C. amplifier with a large amount of negative feedback. The D.C. gain is about 80, as determined by the resistors R_{11} and R_6, and the high frequency response is controlled by the resistor, capacitor network, R_6, C_6.

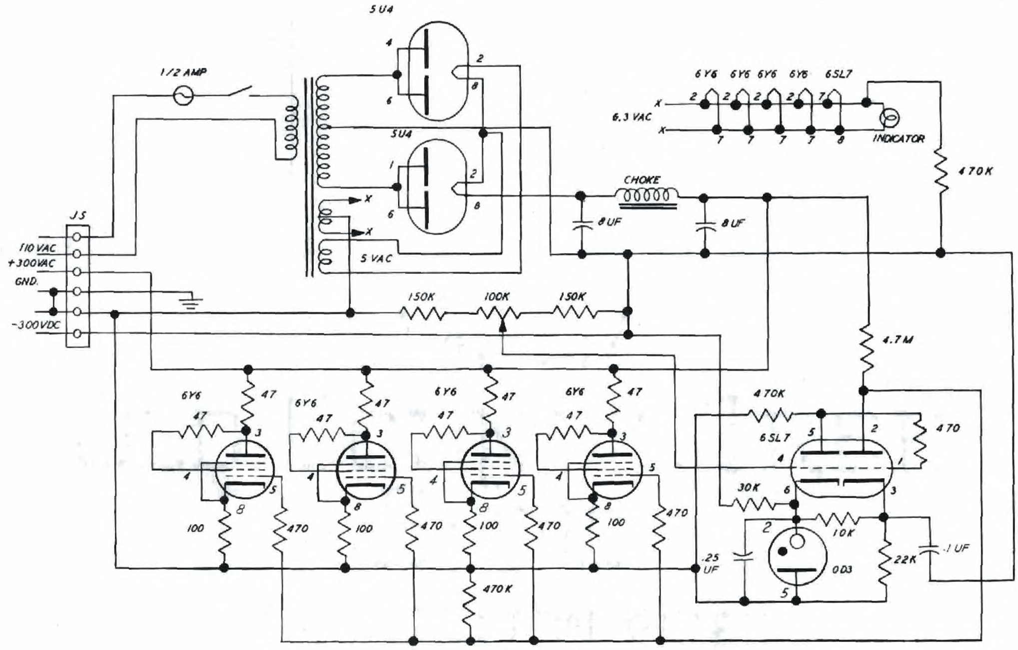
The generator field series regulator is composed of six 6L6-GTB tubes in parallel to carry the 600 milliamperes required by the field. The ten ohm cathode resistors are used to insure proper current division. The 2.2 kilohm resistors in the grid and screen leads are necessary for suppressing parasitic oscillations. The resistors, R_8 and R_9, and the capacitor, C_7, are used to lower the 60 cycle hum output of the rough regulator by shunting the generator field with a low impedance A.C. load.

The resistor, R_7, which is the front panel control labeled Manual Adjust, varies the bias on the 1N2070 diode shunting the output of the operational amplifier. This control is used to vary the generator field current by changing the bias on the 6L6's. This feature allows manual control of the generator output. The manual control is turned full clockwise for automatic operation.

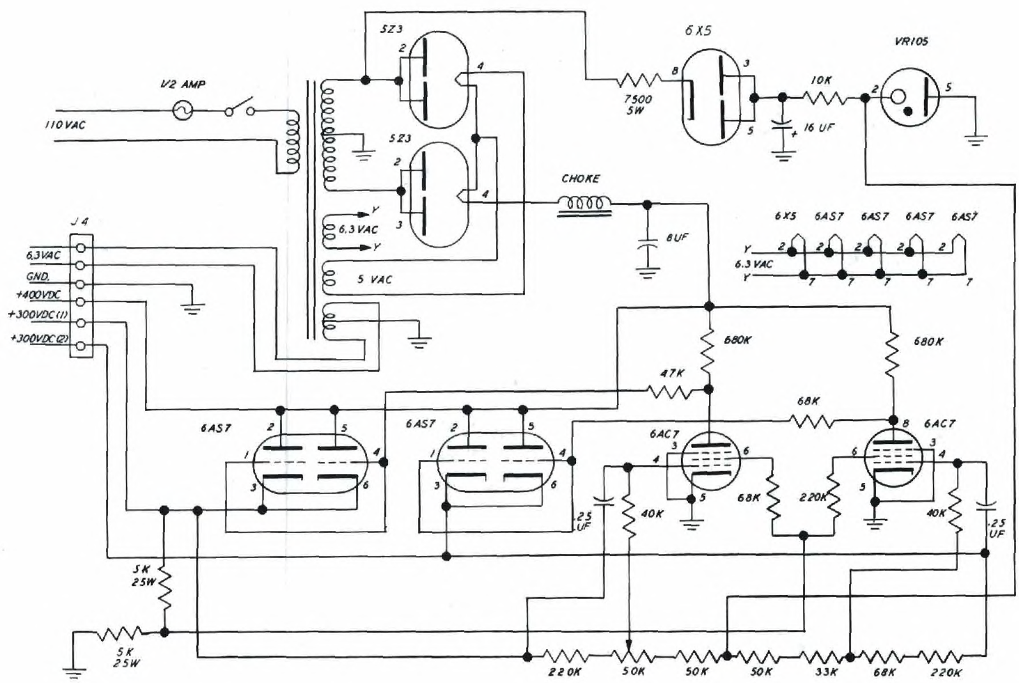
The generator field power supply is a +300 volt, voltage doubler circuit which is capable of delivering up to 0.75 ampere. There is an internal 0.75 ampere fuse in the rough regulator chassis. The A.C. input to this supply is interlocked with a relay operated by the -300 volt bias. Loss of bias will cause the generator field power supply to cut off, reducing the generator output to about one ampere.

There is a 25 volt D.C. supply located in the rough regulator chassis for the filaments of the D.C. amplifier in the fine regulator. This is a simple half-wave rectifier and R-C filter circuit. This circuit is internally fused at 0.5 ampere. The rough regulator chassis contains a 6.3 volts A.C., 6 ampere filament supply also.

The regulated D.C. power supplies are of conventional design. Schematic diagrams of these supplies appear in Figure 7. The -300 volt supply has a floating output and can be used either for positive or negative output. The voltage is adjustable over a small range. The +300 volt supply has two independent +300 volt outputs. Both of the power supplies have more capacity than is needed for the regulator system. The -300 volt unit is capable of delivering approximately 300 milliamperes and the +300 volt units are each capable of delivering about 100 milliamperes. The regulators used about 30 milliamperes from each of the power supplies.



-300 Volt Power Supply



+300 Volt Power Supply

Figure 7. Schematic Diagrams of ±300 Volt Power Supplies.

CHAPTER IV

OPERATION

A picture of the regulator console of the magnet power supply is shown in Figure 8. To turn on the power supply a certain procedure should be followed:

1. Turn on the cooling water.
2. Turn Manual Adjust to the fully counter-clockwise position.
3. Switch the Magnet Power switch to the on position.
4. Set the Current Adjust controls to the minimum position.
5. Push the generator Start button.
6. Turn the Manual Adjust to the fully clockwise position.
7. Set the Current Adjust controls for the desired current.

The magnet power supply turn-off procedure consists of the following steps:

1. Turn the Manual Adjust to the fully counter-clockwise position.
2. Switch the Magnet Power switch to the off position.
3. Push the generator Stop button.
4. Turn the cooling water off.

The Current Adjust knobs are marked in such a manner as to allow a current setting to be repeated very closely. Although the current may be reset from operation to operation by these knobs, the magnetic field is not necessarily the same because of hysteresis and temperature differences.

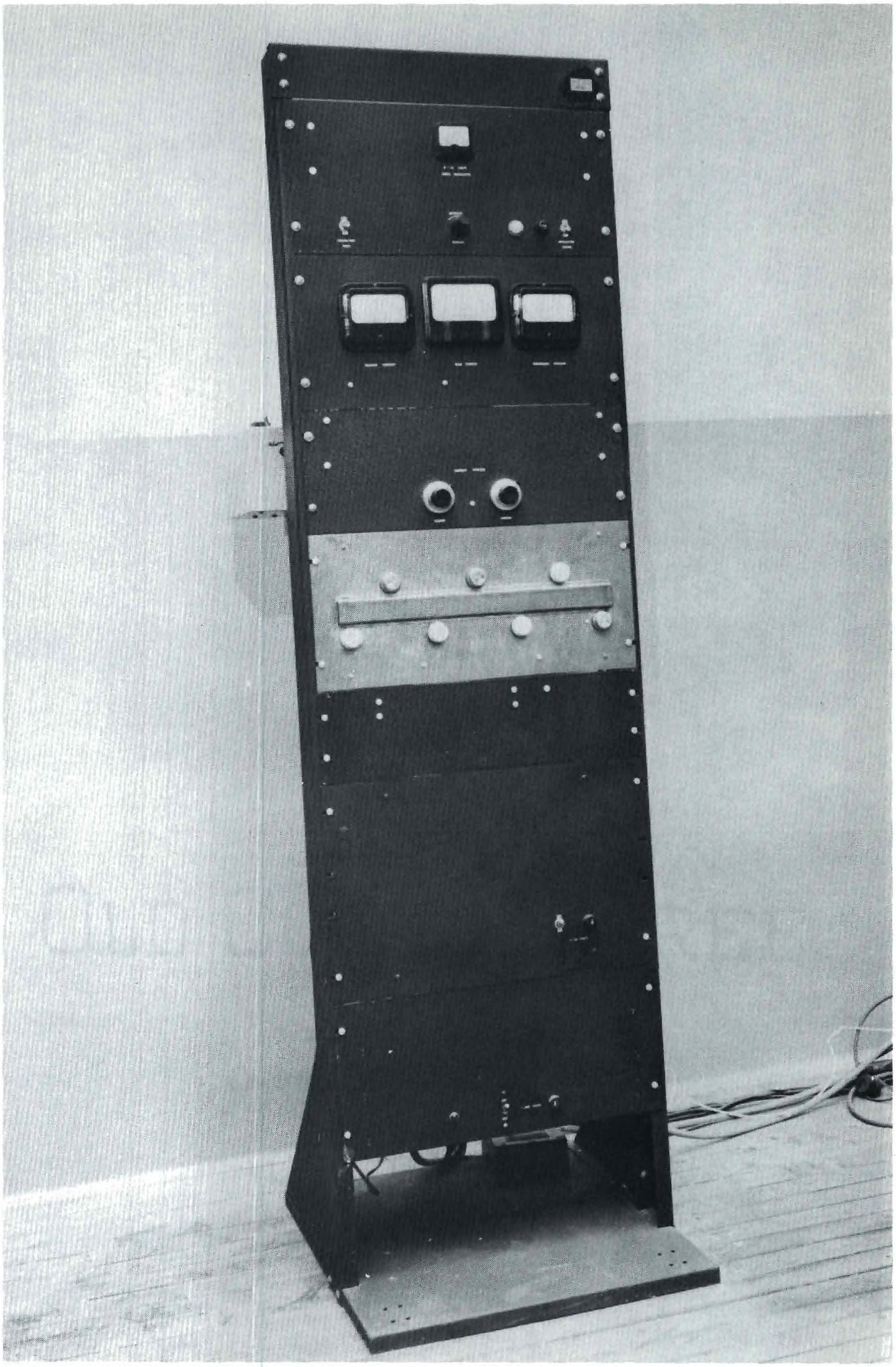


Figure 8. Regulator Console of Magnet Power Supply.

The slow sweep circuit is controlled by an off-on switch and a selector mounted on the side of the regulator rack. The slow sweep has no effect on the regulator when the switch is off. The switch turns on both the drive motor and the sweep reference voltage. The selector is used to choose one of eleven sweep current ranges. The exact current range of any position depends upon the value of the total current flowing in the magnet. There is no observable hysteresis for current sweeps of one per cent or less of the total current.

The turn off procedure is the reverse of the turn on procedure. The generator stop button is pushed and then the Magnet Power switch is turned off. The cooling water should be left on for a few minutes to help dissipate the residual heat.

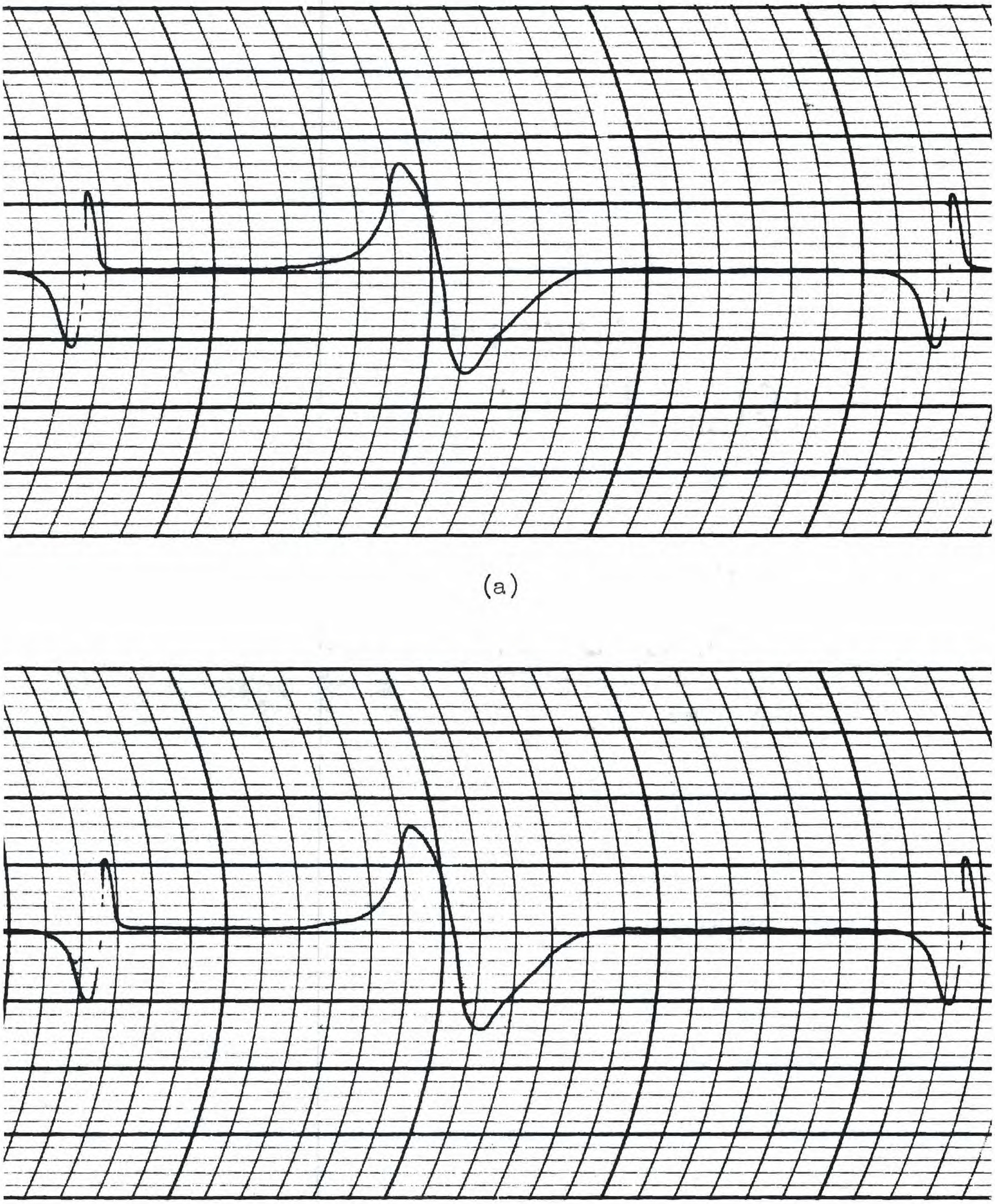
There are seven 1-1/2 volt D-size cells in the regulator. All of these cells have essentially shelf life, but they should be replaced at least every six months. It is recommended that these cells be changed at the beginning of a series of experiments regardless of the previous use or lack of it. One cell is located in the slow sweep box and the other six are located in the fine regulator chassis. They may be reached by removing the side plate and bottom plate, respectively, of the two units. It is important that the correct polarity of the replacement cells be observed.

Figure 9 is a photograph of the nuclear magnetic resonance spectrograph for which the magnet power supply was designed. The power supply short term stability was determined by observing the ripple voltage across the magnet with a high gain, D.C. oscilloscope. The only ripple observable

Figure 9. The Spectrograph.

consisted of a 5 millivolt, peak to peak, 60 cycle component and 20 to 50 millivolt, peak to peak, generator brush noise. The 60 cycle component represents a current ripple of about 0.03 milliamperes. This is approximately one part in 100,000 at the lowest current setting of the power supply. The generator brush noise represents even less current variation as it is at a much higher frequency.

The long term stability of the power supply and of the magnetic field was determined by observation of the absorption line of protons in a 0.5 cubic centimeters sample of water which contained a small amount of ferric nitrate. The natural line width is small and the observed line width is determined primarily by the inhomogeneity of the magnetic field across the sample and by the fluctuations of the field due to power supply variations. The investigation of the long term variation of field consisted of two types of observations. First, the absorption line was observed on the oscilloscope for various values of field, and then the resonance was recorded at a particular field over an extended period of time by means of phase sensitive detector and recorder. There was no observable variation of line shape at any setting of field from about 4,000 gauss to above 10,000 gauss. The observed line width was about 0.3 gauss. The tests were made with constant water flow and relatively constant room temperature. The resonance was recorded by means of the phase sensitive detector and chart recorder. Figure 10 shows examples of the resonance which were taken 10 minutes apart. The large curve is the derivative of the absorption curve. The small peaks mark the beginning of each current sweep and are 1 minute apart in time. The resonance occurred at



(a)

(b)

Figure 10. Recording of Resonant Absorption by Protons in H_2O at Two Times Which are 10 Minutes Apart.

approximately 24.8 megacycles for a field of 5830 gauss. There appears a shift of the resonance line which is about one part in 100,000 per minute after a half-hour of operation. The total change in field in the 10 minute period was about two parts in 100,000. The total variation of field in a five hour period was found to be approximately one part in 100,000.

APPENDIX

CLASSICAL DERIVATION OF THE LARMOR FREQUENCY

Assume a charged spinning body in a uniform magnetic field, $\vec{H}_o$. Further, assume that the body is composed of particles all having the same e/m ratio. The motion of the charges as a result of the rotation of the body constitutes an electric current distribution which can interact with a magnetic field. If the magnetic field is uniform there will be no net force on the body but there will be a net torque

$$\vec{T} = \vec{\mu} \times \vec{H}_o , \tag{39}$$

where $\vec{\mu}$ is the magnetic moment. $\vec{T}$ will also be given by

$$\vec{T} = \frac{d\vec{M}}{dt} \tag{40}$$

where $\frac{d\vec{M}}{dt}$ is the time rate of change of total angular momentum. Combining equations (39) and (40) one gets

$$\frac{d\vec{M}}{dt} = \vec{\mu} \times \vec{H}_o . \tag{41}$$

For any volume distribution of current the magnetic moment is defined as (in Gaussian units)

$$\vec{\mu} = \frac{1}{2c}\int \vec{r} \times \vec{J}\, dV, \tag{42}$$

where $\vec{j}$ is the current density. This equation can be rewritten as

$$\vec{\mu} = \frac{i}{2c} \oint \vec{r} \times d\vec{\ell} \tag{43}$$

if it is assumed $\vec{j}dV = \vec{j}ds d\ell = id\vec{\ell}$ where ds is the cross sectional area of the current and $d\ell$ is an element of length along the direction of current flow. However, $1/2\ \vec{r} \times d\vec{\ell}$ is an element of area swept out by the radius vector and the integral in equation (43) is twice the area, A, of the current loop. If $\vec{n}$ denoted a unit vector normal to the plane of the loop then the magnetic moment is simply

$$\vec{\mu} = \frac{Ai}{c} \vec{n} . \tag{44}$$

Since the body is made up of particles with constant $\frac{e}{m}$ the current density may be expressed

$$\vec{j} = \frac{e}{m} \rho \vec{v} , \tag{45}$$

where ρ is the mass density and $\vec{v}$ is the charge velocity. Substitution of equation (45) into equation (42) gives

$$\vec{\mu} = \frac{e}{2mc} \int \vec{r} \times \rho \vec{v}\, d V . \tag{46}$$

The integral is just the total angular momentum of the body; and therefore, equation (46) becomes

$$\vec{\mu} = \frac{e}{2mc} \vec{M} . \tag{47}$$

The equation of motion can be written

$$\frac{d\vec{M}}{dt} = \vec{M} \times \frac{e}{2mc} \vec{H}_o \ . \tag{48}$$

This is exactly the equation of motion for a vector of constant magnitude which is rotating in space about the direction $\vec{H}_o$ with an angular velocity

$$\vec{\omega}_\ell = - \frac{e}{2mc} \vec{H}_o \ . \tag{49}$$

This is called the Larmor Frequency.

Suppose now that an additional small oscillating magnetic field $\vec{H}'$ is applied at right angles to $\vec{H}_o$ (see Figure 11). Let the oscillating field be

$$\vec{H}' = \vec{H}_1 \cos \omega t \ . \tag{50}$$

The resultant field is

$$\vec{H} = \vec{H}_o + \vec{H}'(t) \ . \tag{51}$$

Since

$$\vec{T} = \dot{\vec{M}} = \frac{e}{2mc} \vec{M} \times \vec{H} \tag{52}$$

dotting $\vec{H}_o$ into both sides gives

$$\left(\frac{d\vec{M}}{dt}\right) \cdot \vec{H}_o = \frac{d}{dt}(\vec{M} \cdot \vec{H}_o) = \frac{e}{2mc} [(\vec{M} \times \vec{H}') \cdot \vec{H}_o + (\vec{M} \times \vec{H}_o) \cdot \vec{H}_o] \tag{53}$$

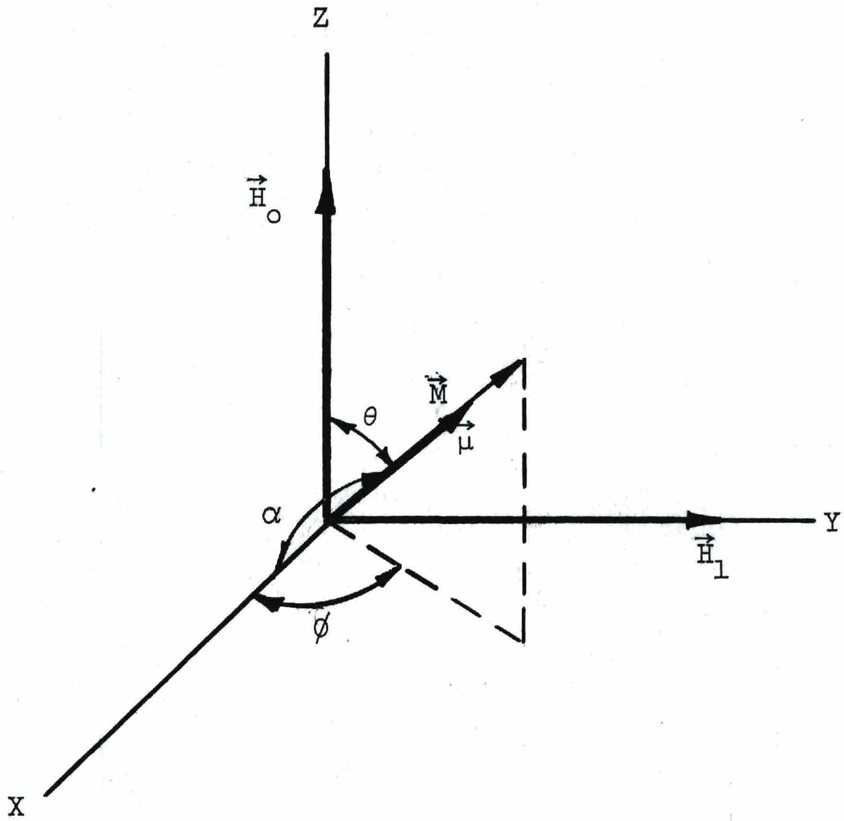


Figure 11. Angular Relationships of $\vec{H}_o$, $\vec{M}$, $\vec{\mu}$, and $\vec{H}_1$.

Since

$$(\vec{M} \times \vec{H}_o) \cdot \vec{H}_o = 0 \tag{54}$$

Equation (53) reduces to

$$\frac{d}{dt}(\vec{M} \cdot \vec{H}_o) = (-MH_o \sin \theta)\dot{\theta} = \frac{e}{2mc} \vec{M} \cdot (\vec{H}' \times \vec{H}_o) \tag{55}$$

or

$$(-MH_o \sin \theta)\dot{\theta} = \frac{e}{2mc} MH'H_o \cos \alpha \tag{56}$$

where α and θ are defined in Figure 11. Dividing Equation (56) by $-MH_o$ one obtains

$$\dot{\theta} \sin \theta = - \frac{e}{2mc} H' \cos \alpha = \omega_K \cos \alpha \cos \omega t \tag{57}$$

where

$$- \omega_K = \frac{e}{2mc} H_1 . \tag{58}$$

From Figure 11 one can obtain the relation

$$\cos \alpha = \sin \theta \cos \phi . \tag{59}$$

Substituting Equation (59) into Equation (57) one gets

$$\dot{\theta} = \omega_K \cos \phi \cos \omega t . \tag{60}$$

If $|\vec{H}_1|$ is very small compared with $|\vec{H}_o|$, then

$$\phi = \omega_\ell t \tag{61}$$

and thus

$$\dot{\theta} = \omega_K \cos \omega_\ell t \cos \omega t \ . \tag{62}$$

Using the trigometric identity

$$\cos A \cos B = \frac{\cos(A+B)}{2} + \frac{\cos(A-B)}{2} \tag{63}$$

one gets

$$\dot{\theta} = \frac{\omega_K}{2} [\cos(\omega_\ell + \omega)t + \cos(\omega_\ell - \omega)t]. \tag{64}$$

Integrating Equation (64) one obtains

$$\theta = \frac{\omega_K}{2} \left[\frac{\sin(\omega_\ell + \omega)t}{\omega_\ell + \omega} + \frac{\sin(\omega_\ell - \omega)t}{\omega_\ell - \omega} \right] \tag{65}$$

Since the limit as ω approaches ω_ℓ of the second term on the right side of Equation (65) is t the value of θ approaches

$$\frac{\omega_K}{4} \left[\frac{\sin 2\omega_\ell t}{\omega_\ell} + 2t \right], \tag{66}$$

and it is evident that θ increases with increasing time when the angular frequency of the time-varying field is equal to the Larmor frequency. This is the classical analogy to the quantum mechanical resonance condition.

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